



Experimental Studies of NaK in a Simulated Space Environment

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Abstract

Space fission power systems are being developed at the National Aeronautics and Space Administration (NASA) and Department of Energy (DOE) with a short term goal of building a full scale, non-nuclear, Technology Demonstration Unit (TDU) test at NASA's Glenn Research Center. Due to the geometric constraints, mass restrictions, and fairly high temperatures associated with space reactors, liquid metals are typically used as the primary coolant. A eutectic mixture of sodium (22 percent) and potassium (78 percent), or NaK, has been chosen as the coolant for the TDU with a total system capacity of approximately 55 L. NaK, like all alkali metals, is very reactive, and warrants certain safety considerations. To adequately examine the risk associated with the personnel, facility, and test hardware during a potential NaK leak in the large scale TDU test, a small scale experiment was performed in which NaK was released in a thermal vacuum chamber under controlled conditions. The study focused on detecting NaK leaks in the vacuum environment as well as the molecular flow of the NaK vapor. This paper reflects the work completed during the NaK experiment and provides results and discussion relative to the findings.

Introduction

The experimental parameters and test scenarios associated with this experiment are best understood by having some background information on the TDU test (Fig. 1). The general concept involves pumping NaK through the TDU test loop where thermal energy is added at the reactor simulator and removed at the Power Conversion Unit (PCU). The PCU uses the thermal energy supplied from the loop to power its thermodynamic cycle, resulting in the overall goal of electrical power. The PCU waste heat is transferred to water and rejected through the 6 radiator panels that make up the Heat Rejection System (HRS). At nominal TDU test conditions the heat addition is 48 kW_e at a nominal outlet temperature of 875 K, power conversion is 12 kW_e, and heat rejection is 36 kW_t.

Although the NaK fluid piping and system components will be rigorously designed, the ability to detect, manage, and clean up a NaK leak will be important for proper test preparation. To study this, an experiment was designed and performed in a smaller facility where cleanup and potential facility damage would be limited. The key topics of the experiment were: detection instrumentation and methods, molecular flow of NaK, and post leak processes and procedures.

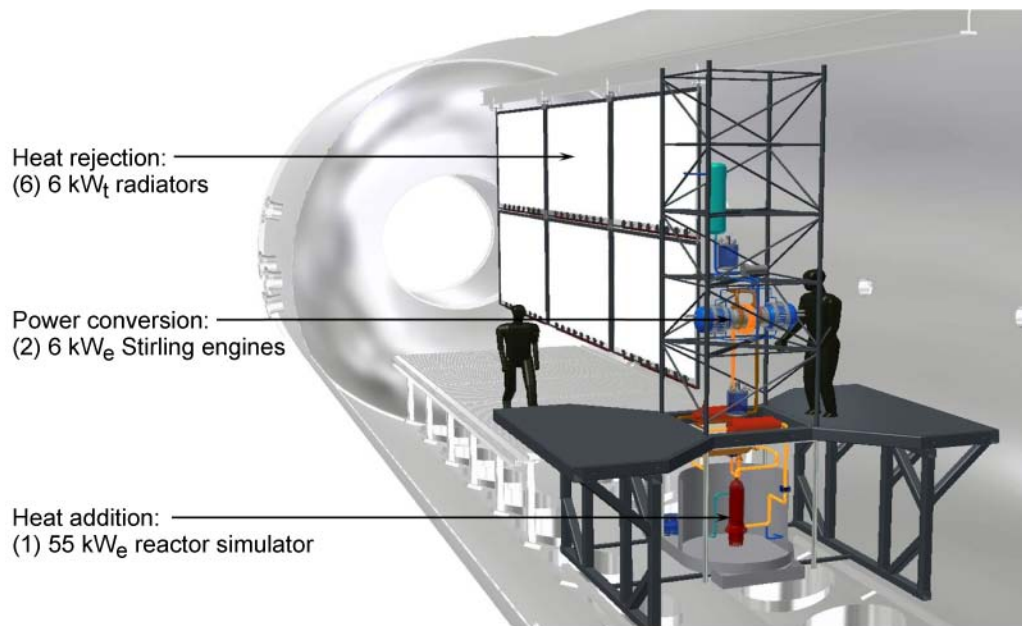


Figure 1.—TDU test layout.

Nomenclature

NASA	National Aeronautics and Space Administration
DOE	Department of Energy
VF6	vacuum facility 6
MLI	multilayer insulation
TDU	Technology Demonstration Unit
NaK	A eutectic mixture of sodium and potassium
NaOH	sodium hydroxide
KOH	potassium hydroxide
HRS	Heat Rejection System
GN_2	gaseous nitrogen
K	Kelvin
μg	micro grams
amu	atomic mass units
L	Liter
\AA	Angstrom ($1 \times 10^{-10}\text{ m}$)
AT	Alpha Temperature
SEM	scanning electron microscopy
RGA	Residual Gas Analyzer
QCM	Quartz Crystal Microbalance

Experiment Design

The simulated space environmental requirements were established in the TDU test specification with a pressure of 1.0×10^{-6} torr and an effective thermal sink of 175 K. Vacuum facility 6 at the Glenn Research Center (GRC) has been chosen for the TDU test because of its physical size and thermal environment. In order to keep the cost and risk assessment of this experiment low, a smaller scale facility with comparable characteristics to VF6 was needed. Vacuum facility 17, also located at GRC, was chosen because its thermal vacuum design is similar to VF6, allowing a small scale experimental setup.

Based on the vapor pressure curve of NaK (Borgstedt & Mathews, 1987), boiling will occur at 336 and 364 K at the respective pressures of 1.0×10^{-6} and 1.0×10^{-5} torr as seen in Figure 2. During the TDU testing, the NaK will have a nominal operating temperature of 875 K which will boil at 75 torr. As the results will show, the pressure of NaK during a leak event is difficult to measure. The difference between the boiling point and the temperature of the leaking NaK at any given pressure will determine the rate at which vaporization occurs. The planned use of multilayer insulation (MLI) in the TDU will complicate the ability to measure the temperature and pressure of the potential leak and therefore minimize prediction accuracy on vaporization rates. Understanding this issue, the experiment was designed to study low vaporization rates of NaK leaks inside a thermal vacuum environment and determine our ability to measure and quantify the event. In order to accomplish this task, appropriate detection sensors and methods were needed.

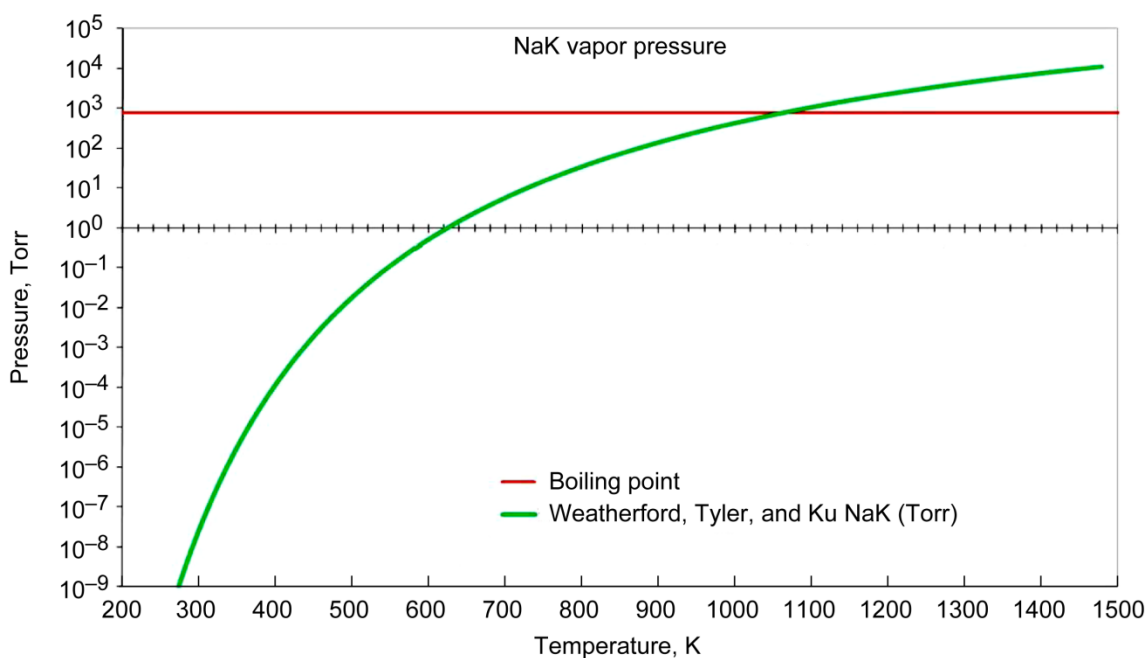


Figure 2.—Vapor pressure curve.

NaK Detection Sensors

Several different instruments were reviewed to determine which hardware would be best suited for detecting a NaK leak. At completion of the evaluation it was determined that a Quartz Crystal Microbalance (QCM) and a Residual Gas Analyzer (RGA) were the best candidates for near term evaluation.

The Alpha Temperature (AT) cut gold plated quartz crystal has a resonant frequency of 6 MHz with an effective deposition area of 55 mm². A specialized controller and oscillator enable the system to monitor the changes in frequency of the crystal. This change in frequency is proportional to the deposition mass and is displayed as a film thickness. This method provides measurement accuracy of 0.5 percent to a level of less than 1.0 Å. This technology is widely used in the semiconductor and material coating industry but has not been used for detecting NaK deposition in a space simulation environment.

The quadrupole residual gas analyzer identifies and measures the partial pressure of gases up to 200 amu. The ability to identify the Na, K, and the respective reactants separately provides detailed information needed for analysis. In speaking with different RGA manufacturers it was found that very few had experience measuring sodium or potassium. Consequently, as with the QCM, this test would provide new information regarding measuring Na and K in a space simulated environment.

Test Apparatus

A view of the test apparatus (Fig. 3) depicts the necessary components needed to perform and evaluate a controlled release of NaK inside a vacuum chamber. Starting at the bottom, the NaK is contained in the sample cylinder by manual valve MV-1. During initial atmospheric operations, the remote operated valve ROV-1 is opened prior to MV-1 to provide a GN₂ purge. After achieving rough vacuum (10⁻³ torr), the GN₂ is shut off, the chamber continues into high vacuum (10⁻⁶ torr), and the cold wall is filled. Once at test conditions, the NaK is heated to boiling, allowing the vapor to flow across the

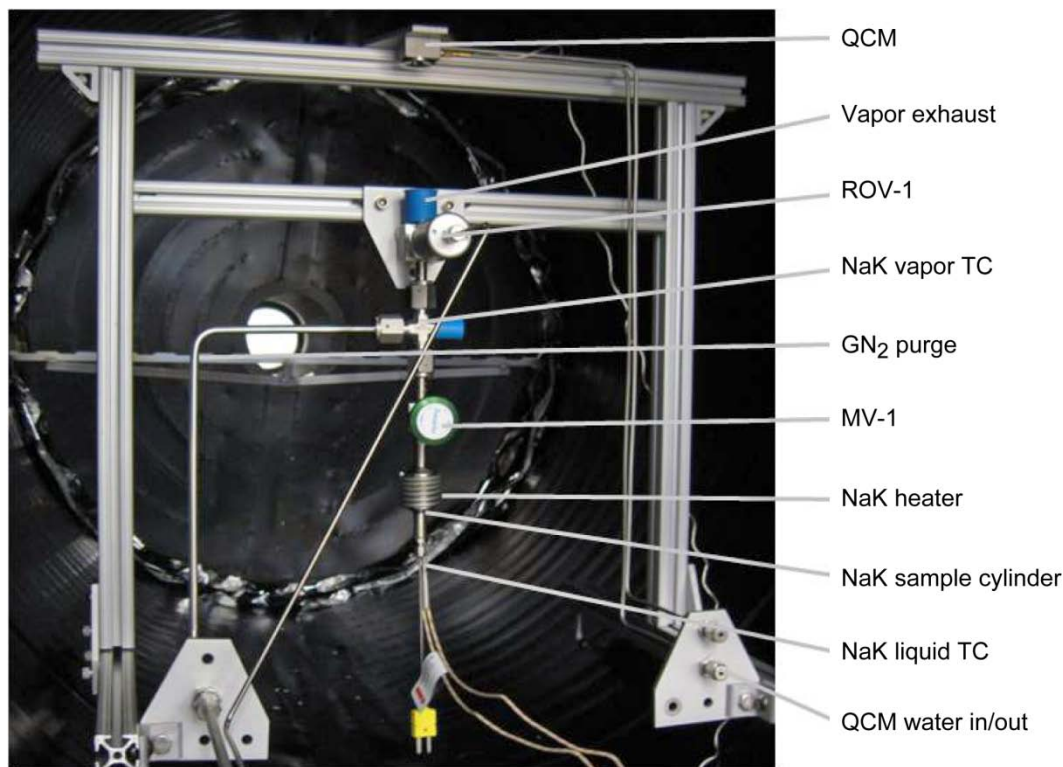


Figure 3.—Test apparatus.

vapor thermocouple (TC), through ROV-1, and out the exhaust port. At this point, the NaK vapor is in free molecular flow where it can move throughout the chamber and be detected with the QCM and RGA instrumentation. The RGA, not seen in this picture, was located outside of the pressure vessel with a sensing port at the front section of the chamber, just behind the cold wall. The QCM was located at the top of the test stand.

Results and Discussion

Many tests were completed to evaluate the detection abilities of the two instruments and determine their effectiveness for use in the TDU system test. Figure 4 illustrates test results in which boiling of the NaK occurred and the instrumentation recorded the event. The QCM data including NaK deposition thickness and temperatures can be viewed in the top graph while the RGA data is detailed in the lower graph. The X axis has been synchronized with both sets of data for real-time comparisons.

The data can best be analyzed by breaking the time scale into separate events (1–6). The NaK heater was started at event 1 by adjusting power through a Variac transformer. Adjustments to the heater power can be seen throughout the testing as more power was needed to increase the temperature.

At event 2, the upper QCM graph shows vapor being deposited on the crystal when the NaK reached 423 K (QCM, blue). Between events 2 and 3 the QCM had measured a deposition of 10\AA of NaK at a rate of 0.3 \AA/min . On the lower RGA graph, Na appears, becoming increasingly present at the 1×10^{-9} scale as the NaK approached its boiling point. During this time frame, an increase in H (2, blue) was present which was believed to be related to the reactions taking place at the cold wall between the Na (23, red) and H_2O (18, black). The change in hydrogen pressure was easy to see on the RGA in relation to the water vapor. This is mainly due to the fact that the hydrogen could not be pumped by the cold wall, making it easy to reach the RGA inlet port outside of the cold wall enclosure.

The sample cylinder continued to be heated to 648 K which brought the NaK to full boiling at event 3. This event is best illustrated by looking at the vapor temperature (QCM, purple) in the upper graph as the slope changes from 381 to 450 K. The rate of deposition on the QCM also changed slope during this time frame with a maximum rate of 1.0 \AA/min . The RGA continued to detect H along with Na during the beginning stages of boiling but did not provide definitive results in detecting K (39, purple). This is most likely related to the fact that potassium's atomic mass is fairly close to Ar (40) and NaOH (40) making it ambiguous as a trigger event. Sodium, which has an atomic mass of 23, happens to fall in a fairly void section of the detection spectrum making it easier to identify as a trigger event.

The NaK continued to boil between events 3 and 5 until most of it had been vaporized in the capsule. Event 4 shows the point at which the liquid had dropped below the NaK liquid TC tip giving an offset between the liquid temperature and the capsule temperature.

At event 5 the heaters were turned off and the system was allowed to cool. The NaK deposition on the QCM stopped shortly after event 5 but continued to register on the RGA. Due to the location of the RGA, it is probable that the Na molecules were trapped between the warmer chamber shell and outer cold wall until they found their way out the pumping train. This phenomenon was more drastic during other tests in which the cold wall was off and incapable of cryopumping the Na molecules. As the system continued to cool the Na was pumped by either mechanical means or by the cold wall as can be seen at event 6.

The RGA showed this type of lagging behavior in many of the performed tests. It became apparent that the detection of Na with the RGA was time dependent in relation to the test hardware and pumping train locations. The RGA's line of sight to the test hardware would prove to be an important variable for determining its effectiveness as a sensor. The location of the RGA for this experiment in VF17 was a worst case scenario with the sensing port located behind the cold wall facing the MLI. This created a bounce space for the Na molecules, which given the right conditions, would create a time lag between the leak point and detection point. In VF6, the RGA has an opening through the cold wall allowing this localizing effect to be minimized. This characteristic led to uncertainty with the RGA's ability, by itself, to quickly and correctly identify a NaK leak.

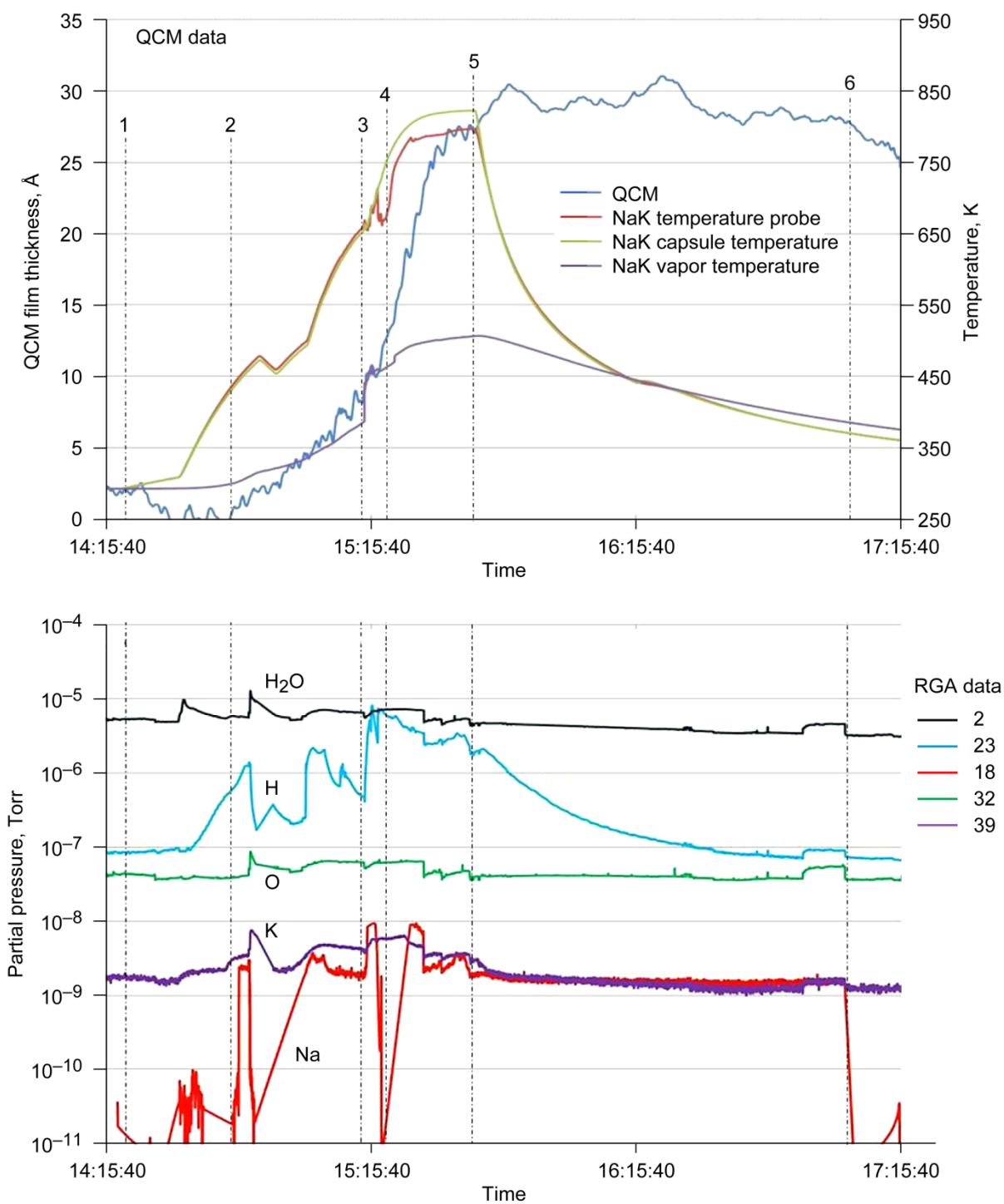


Figure 4.—NaK boiling event: NaK deposition on QCM crystal versus temperature (upper), RGA detection (lower).

The QCM was able to measure very small depositions of NaK as well as determine the rate at which the deposition was occurring. This capability can be used to determine the start of a leak event as well as quantify the potential size of a leak by monitoring the deposition rate. This characteristic of the QCM proved to be valuable when trying to determine whether the NaK in the chamber was residual from prior releases or if there was new vaporization taking place. Additionally, the ability of the QCM to be located in close proximity to the test hardware allowed the sensor to give definitive results on real-time deposition and contamination. This left very little confusion on whether there was a contamination event taking place. Another characteristic of the QCM that needs emphasis is the pressure range at which the sensor can be used. The QCM can work in atmospheric pressures and under high vacuum as long as it is being actively cooled by chilled water. This could prove useful during testing in which the tank pressure is above 1×10^{-5} torr; the RGA upper pressure limit. The only real negative to the QCM is its inability to identify the material that is depositing on its surface. This issue could trigger false positives during testing leading to incorrect shutdown procedures. Due to this characteristic, the QCM, by itself, is limited in providing definitive results on a NaK leak.

Post-Test Analysis

Follow-on diagnostics of the tested QCM crystals using electron microscopy was performed to determine the presence of NaOH or KOH. Figure 5 shows pictures of the QCM at the completion of the test using SEM imaging. The NaK deposited discs in pictures “a” and “b” are clearly visible with the outer, lighter ring being protected from the deposition by the crystal holder. Pictures “c” and “d” show the SEM images of the crystal with the gold flakes and the deposited NaK, now hydroxides from post test exposure with air.

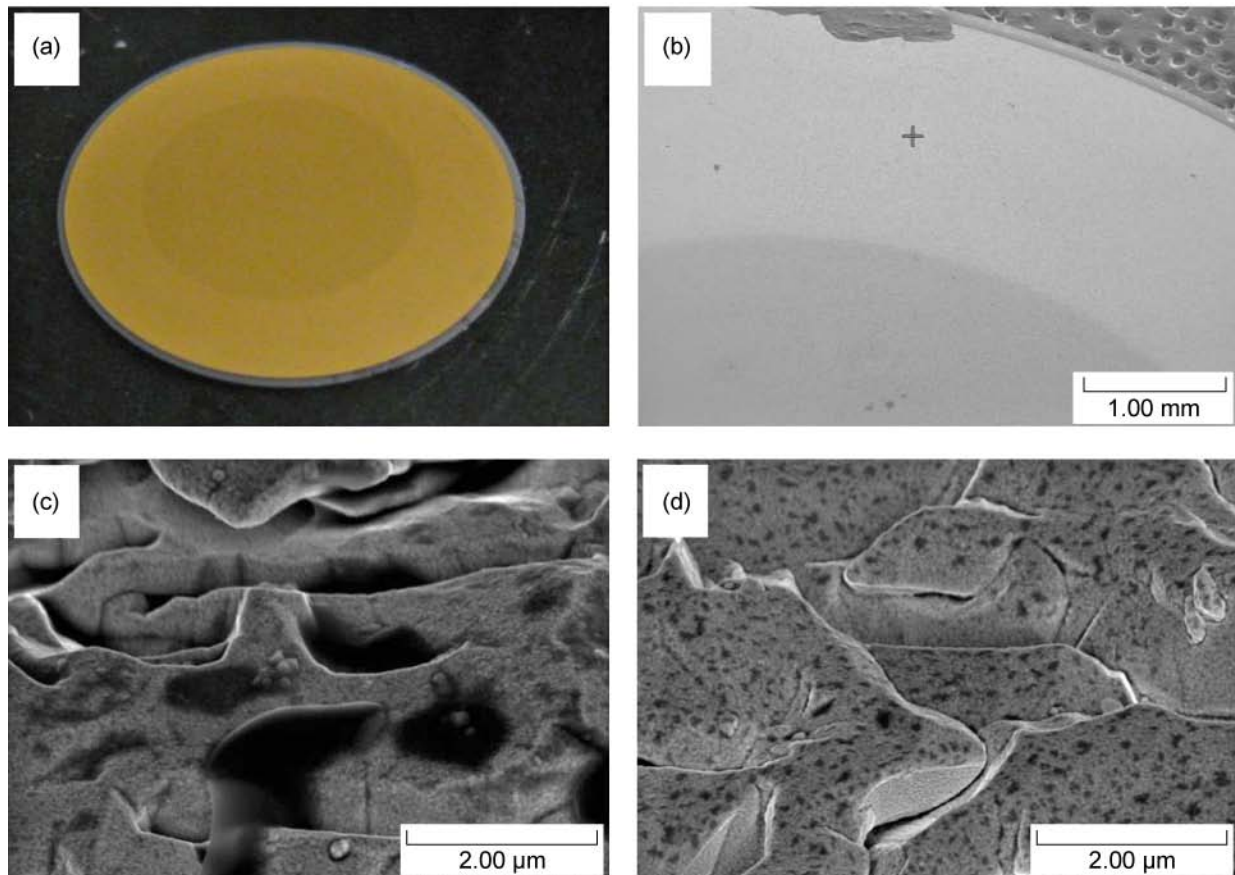


Figure 5.—QCM images.



Figure 6.—Globular NaK deposits on VF17 cold wall.

Additional analysis involved studying NaK deposition locations. During the testing, approximately 7 mL of NaK was released into the chamber with two types of deposits; the vaporized deposition as discussed above, and the globular deposits, seen in Figure 6. The globular deposits were located at the 6:00 and 12:00 middle positions where the NaK would “burp” during heating. This behavior is believed to be caused by vapor bubbles trapped between fluid layers.

The location and concentration of the vaporized deposits were studied to understand how the NaK molecules would travel around the chamber. With this information, it was deduced that the NaK deposition thickness had a direct relationship to its directional velocity vector or “line of sight.” The NaK vapor was found to be easily pumped by cool surfaces, leaving a concentration map that related to the vapor’s geometric origins.

Post test experimentation with pressurizing the chamber after a NaK contamination was studied to develop safe practices. As a trial procedure, the incoming atmosphere was restricted during pressurization to minimize the rate of reaction between the water vapor and NaK, reducing the amount of caustic fumes that are generated. This was accomplished by letting small quantities of air and GN_2 into the chamber until smoke could be seen in the view ports. At this time, the vent valve was closed, allowing the NaK to react with the controlled quantity of air. This allowed the globular NaK deposits to slowly react with the small quantities of water vapor from the atmosphere and form the less reactive NaOH and KOH. This method took approximately 8 hrs to complete and reduced the NaK into NaOH and KOH which were much safer and easier to clean up. This method could be used in conjunction with the vacuum facility pumping train to allow fresh quantities of mixed GN_2 and air into and out of the chamber during pressurization. This type of gas recycling during vent up could be used to transfer the caustic fumes out of the facility and provide a safer cleanup scenario.

Although this pressurization procedure produced successful results during this small scale leak experiment it is unsure whether or not a larger cleanup would require more time to complete. Longer exposures of NaK in oxygen create a problem with the potential formation of potassium super oxide (KO₂). The exact, time dependent formula for KO₂ formation is unknown and therefore presents uncertainties about how long the NaK can be exposed to oxygen before cleanup takes place. Further analysis is needed to help determine whether or not large scale cleanups could follow this trial method of pressurization. These results along with additional studies will be used to reduce the risks associated with the TDU test and improve safety of the facility and personnel.

Conclusions

The test results have given conclusive data that detection of NaK in a space simulated environment can be achieved using both the QCM and RGA methods together. Using sodium as the signature atomic mass, the RGA will be the primary detector with a partial pressure range down to 1×10^{-10} torr. Quantifying the amount of NaK being released can be accomplished using the QCM as a deposition thickness and rate monitor. Together, these instruments should provide enough data to successfully detect a NaK leak and provide opportunity to use precautionary procedures and minimize any potential damage to the facility and test hardware.

The NaK vapor deposition during a leak event will typically accumulate on the first cold surface it encounters. The rate of vaporization and direction will determine where the NaK vapor ends up inside the vacuum chamber while the surface temperature of the chamber will determine whether the vapor deflects or deposits on the surface. In relation to the TDU test, the MLI over the NaK containment piping should reduce any vapor jet streams from focusing the NaK in any particular direction in the vacuum chamber. If the NaK vapor escapes the MLI before condensing, it would be less concentrated and directional in its path, thus producing less deposition on the chamber surfaces. This type of scenario would be detected using the RGA and QCM and allow a safe shutdown procedure prior to analyzing where the leak originated.

Methods for pressurizing a vacuum chamber after NaK contamination have been studied using a combination of air and gaseous nitrogen. Slowly allowing the NaK to mix with known quantities of air and GN₂ allows control of the reaction rate between NaK and atmospheric water vapor. Recycling of the gas mixture inside the vacuum chamber can be done with rough pumps and or ventilation valves to try and remove any harmful hydroxide fumes associated with the NaK and water vapor reactions. More work is needed to verify if this method would be appropriate for the TDU and VF6.

The results and procedures developed from this experiment have given clarity and understanding involving the detection and molecular flow of NaK in a space simulated environment. These results will be used to reduce the risks associated with the safety of the TDU test hardware, facility, and personnel.

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